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NICOTINE INSECTICIDES. PART I--NEW METAL-NICOTINE COMPOUNDS

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Twenty-five nicotine compounds of a water-resistant type, in which the nicotine is combined usually with a metal to form a complex salt, were prepared in 1942 and 1943 at the Eastern Regional Research Laboratory of the Bureau of Agricultural and Industrial Chemistry, for testing against plant-feeding insects by the Bureau of Entomology and Plant Quarantine at its Sanford, Fla., laboratory. Previous work by Hansberry 2/ on a few of these materials had shown that as stomach insecticides they were more toxic to newly hatched larvae of the codling moth (Carpocapsa pomonella (L.)) than were the soluble compounds tested by him. The tests reported herein were made to determine insect mortality by stomach and contact action, as well as the effect of spray residues and dust deposits left on foliage. The testing methods are somewhat similar to those recently described by Swingle 3/.

Chemical Nature of Metal-Nicotine Salts

The direct combination of nicotine with an acid results in the formation of a simple salt. Most, if not all, of these simple salts are very soluble in water. Certain ones, such as the salicylate, benzoate, tartrate, and oxalate, are crystalline solids, stable under atmospheric conditions but soluble in water. Many other simple salts are of indefinite composition, and usually hygroscopic. The water-resistant combinations of nicotine with bentonite, tannic acid, or humic acid might be considered as simple salts, but the resemblance is probably superficial.

1/ Now assistant entomologist, Division of Insects Affecting Man and Animals.

2/ Hansberry, R. Toxicity of nicotine compounds to newly hatched codling moth larvae. Jour. Econ. Ent. 35: 915-918. 1942.

3/ Swingle, M. C. Exploring the insecticidal possibilities of new materials. In Laboratory procedures in studies of the chemical control of insects, edited by F. L. Campbell and F. R. Moulton, Amer. Assoc. Adv. Sci. 20, pp. 82-84. Washington, D. C. 1943.

A study of the toxicity of the more complex metal-nicotine salts is important in determining whether the toxicity of nicotine is influenced favorably or otherwise by its presence in chemical union with the various metals employed. Our knowledge of the effect of molecular structure on toxicity is inadequate. Furthermore, resistance to weathering, including moisture and oxidation effects, would seem to be a desirable property.

The complex salts of nicotine with a selected metal are of two types, the double salt and the nicotinammino compound. The former results from the combination of the metal and nicotine salts of the same selected acid. The latter is formed when the nicotine alkaloid reacts with a metal salt of the selected acid. The metals most likely to form these two types of compounds are those that are less electropositive than magnesium, such as iron, cobalt, nickel, copper, cadmium, manganese, zinc, aluminum, and chromium. Of the acids so far tried, the most suitable for forming complex salts that are relatively insoluble in water are benzoic, salicylic, picric, thiocyanic, hydrocyanic, o-benzoylbenzoic, and o-phenoxybenzoic.

The compounds tested, with their formulas, are listed below. It will be noted that (10), (11), and (12) contain no metal but are salts of dye acids (13), (14), and (15) are the corresponding metal-nicotine double salts of the dye acids 4/.

- (1) Cupric dinicotine thiocyanate, $\text{Cu}(\text{CNS})_2 \cdot 2 (\text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HCNS})$.
- (2) Cupric dinicotine o-benzoylbenzoate,
 $\text{Cu}(\text{OOC} \cdot \text{C}_6\text{H}_4 \cdot \text{OC} \cdot \text{C}_6\text{H}_5)_2 \cdot 2 (\text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HCOO} \cdot \text{C}_6\text{H}_4 \cdot \text{OC} \cdot \text{C}_6\text{H}_5)$
- (3) Cupric dinicotine benzoate, $\text{Cu}(\text{OOC} \cdot \text{C}_6\text{H}_5)_2 \cdot 2 (\text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HOOC} \cdot \text{C}_6\text{H}_5)$.
- (4) Cupric dinicotine salicylate monohydrate,
 $\text{Cu}(\text{OOC} \cdot \text{C}_6\text{H}_4(\text{OH}))_2 \cdot 2 (\text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HOOC} \cdot \text{C}_6\text{H}_4(\text{OH})) \cdot \text{H}_2\text{O}$.
- (5) Zinc mononicotine thiocyanate, $\text{Zn}(\text{CNS})_2 \cdot \text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HCNS}$.
- (6) Cupric dinicotine picrate,
 $\text{Cu}(\text{O} \cdot \text{C}_6\text{H}_2(\text{NO}_2)_3)_2 \cdot 2 (\text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HO} \cdot \text{C}_6\text{H}_2(\text{NO}_2)_3)$.
- (7) Zinc dinicotine picrate,
 $\text{Zn}(\text{O} \cdot \text{C}_6\text{H}_2(\text{NO}_2)_3)_2 \cdot 2 (\text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HO} \cdot \text{C}_6\text{H}_2(\text{NO}_2)_3)$.
- (8) Zinc dinicotine salicylate,
 $\text{Zn}(\text{OOC} \cdot \text{C}_6\text{H}_4(\text{OH}))_2 \cdot 2 (\text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HO} \cdot \text{C}_6\text{H}_4(\text{OH}))$.

4/ Compounds (12) to (15), inclusive, were prepared by C. W. Murray, of the Bureau of Agricultural and Industrial Chemistry.

- (9) Zinc dinicotine benzoate, $\text{Zn}(\text{OOC} \cdot \text{C}_6\text{H}_5)_2 \cdot 2(\text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HCOOC} \cdot \text{C}_6\text{H}_5)$.
- (10) Nicotine chrome orange R, salt of p-nitrobenzeneazosalicylic acid, the dye acid of chrome orange R.
- (11) Nicotine paper yellow L (first fraction), salt of 2, 4-dinitro-1-naphthol.
- (12) Nicotine paper yellow L (second fraction), similar to (11) but exact constitution unknown.
- (13) Cupric nicotine chrome orange R, double salt with the dye acid of (10).
- (14) Cupric nicotine paper yellow L, double salt with the dye acid of (11).
- (15) Cupric nicotine stilbene yellow GA, double salt with the acid of a sulfonated nitrostilbene dye.
- (16) Cuprous mononicotine thiocyanate, $2 \text{CuCNS} \cdot \text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HCNS}$.
- (17) Cuprous dinicotine thiocyanate, $2 \text{CuCNS} \cdot 2(\text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HCNS})$.
- (18) Cobaltous dinicotine thiocyanate, $\text{Co}(\text{CNS})_2 \cdot 2(\text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HCNS})$.
- (19) Nickelous dinicotine thiocyanate, $\text{Ni}(\text{CNS})_2 \cdot 2(\text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HCNS})$.
- (20) Cuprous nicotine cyanide, $2 \text{CuCN} \cdot \text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HCN}$.
- (21) Manganous mononicotine salicylate,
 $\text{Mn}(\text{OOC} \cdot \text{C}_6\text{H}_4(\text{OH}))_2 \cdot \text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HOOC} \cdot \text{C}_6\text{H}_4(\text{OH})$.
- (22) Cadmium dinicotine salicylate,
 $\text{Cd}(\text{OOC} \cdot \text{C}_6\text{H}_4(\text{OH}))_2 \cdot 2 \text{C}_{10}\text{H}_{14}\text{N}_2 \cdot \text{HOOC} \cdot \text{C}_6\text{H}_4(\text{OH})$.
- (23) Cupric dinicotinammino benzoate dihydrate,
 $\text{Cu}(\text{OOC} \cdot \text{C}_6\text{H}_5)_2 \cdot 2 \text{C}_{10}\text{H}_{14}\text{N}_2 \cdot 2\text{H}_2\text{O}$.
- (24) Cupric dinicotinammino o-benzoylbenzoate dihydrate,
 $\text{Cu}(\text{OOC} \cdot \text{C}_6\text{H}_4(\text{OC} \cdot \text{C}_6\text{H}_5))_2 \cdot 2 \text{C}_{10}\text{H}_{14}\text{N}_2 \cdot 2\text{H}_2\text{O}$.
- (25) Cuprous dinicotinammino thiocyanate, $(\text{CuCNS})_2 \cdot 2 \text{C}_{10}\text{H}_{14}\text{N}_2$.

The insecticide used for comparison in most of the tests was that usually recommended for control of the species. These materials were pyrethrum (pyrethrin I 0.60 percent and pyrethrin II 0.63 percent), derris (rotenone 4.8 percent), barium fluosilicate, nicotine sulfate, lead arsenate, sodium fluoride, and dusting sulfur. In some cases they were applied at a greater strength than is recommended for field use.

The tests were made on representatives of five orders of insects and a red spider as listed below, with the host plants and form used for each.

INSECT	HOST
Orthoptera (large nymphs): American cockroach (<u>Periplaneta americana</u> (L.))	- - - - -
Isoptera (adults and large nymphs): A termite (<u>Reticulitermes</u> sp.)	- - - - -
Homoptera (wingless agamic form): An aphid (<u>Macrosiphum ambrosiae</u> (Thos.))	Grassleaf wild lettuce (<u>Lactuca graminifolia</u>)
Coleoptera (adults): A blister beetle (<u>Epicauta lemniscata</u> (F.))	Figweed (<u>Amaranthus</u> sp.)
Cowpea weevil (<u>Callosobruchus maculatus</u> (F.))	Brown Crowder cowpea (<u>Vigna sinensis</u>)
Lepidoptera (fourth instars): Bean leaf roller (<u>Urbanus proteus</u> (L.))	Bean (<u>Phaseolus vulgaris</u>)
Cross-striped cabbage worm (<u>Evergestis rimosalis</u> (Guen.))	Collard (<u>Brassica oleracea acephala</u>)
Greenhouse leaf tier (<u>Phlyctaenia rubricalis</u> (Guen.))	do.
Hawaiian beet webworm (<u>Hymenia fascialis</u> (Gram.))	Figweed (<u>Amaranthus</u> sp.)
Melon worm (<u>Diaphania hyalinata</u> (L.))	Pumpkin and squash (<u>Cucurbita</u> spp.)
Pickleworm (<u>Diaphania nitidalis</u> (Stoll.))	do.
Southern beet webworm (<u>Pachyzancla bipunctalis</u> (F.))	Figweed
Southern armyworm (<u>Prodenia eridania</u> (Gram.))	Collard, pigweed
Acarina (all stages): Common red spiders (<u>Tetranychus</u> sp.)	Celery (<u>Apium graveolens</u>)

Eleven kinds of plants were used in the tests for phytotoxicity — bean, broccoli, collard, lettuce, okra, pea, potato, pumpkin, swiss chard, tomato, and turnip. An attempt was made to use at least five kinds with each concentration of the chemicals, but since the tests were made at different times of the year it was impossible to use the same five for all the compounds.

Tests with Dusted Foliage

The first experiment was to determine the efficiency of the compounds as dusts. The material was applied to both sides of leaf sections in a settling chamber, and the approximate deposit determined from the weight of the dust on a small metal plate placed in the chamber with the leaves. Two dusted leaf sections were used for each species of insect, each leaf section being placed with 15 insects in a 9-cm. Petri dish and held at a constant temperature of 80° F. Mortality counts and estimates of the amount of feeding were made at the end of 2 and 3 days. The mortality in these closed dishes may have been due to either stomach or contact action. Since these tests were only preliminary, no replications were made.

The compounds of high nicotine content when used as undiluted dusts were very toxic to nearly all the test insects, but the results with only the more practical dilutions are reported here. From the data acquired it appeared that six of the compounds (table 1) were more effective than the others at dilutions containing approximately 5 percent of nicotine. It should be noticed that four of these six materials contained copper as the metal. The blister beetle, the greenhouse leaf tier, and the southern armyworm were shown to be very resistant to all compounds tested. The bean leaf roller and the pickleworm were very susceptible, and the cross-striped cabbage worm, the Hawaiian beet webworm, the melon worm, and the southern beet webworm moderately susceptible.

Table 1.--- Toxicity of the 6 best of 25 metal-nicotine compounds tested as compared with recommended insecticides when the insects were fed dusted foliage

Compound and insect	Nicotine content of dust	Deposit of dust per square centimeter	Period of exposure	Extent of feeding	Mortality
	Percent	Micrograms	Days		
Cupric dinicotine picrate:					
Hawaiian beet webworm	6.0	200	3	Moderate	89
Melon worm	5.0	195	3	---do---	90
Pickleworm	5.0	190	3	None	100
	2.5	195	3	Trace	100
Southern armyworm	6.0	210	3	Much	3
Southern beet webworm	6.0	210	3	Trace	71
	2.4	210	3	Much	43
Zinc dinicotine picrate:					
Hawaiian beet webworm	12.0	200	3	Trace	86
Melon worm	5.0	200	3	Moderate	90
Pickleworm	5.0	200	2	None	100
	2.5	190	2	Moderate	100
Southern armyworm	6.0	200	2	Much	8
Southern beet webworm	6.0	195	3	Moderate	90
	2.4	200	2	Much	0
Cuprous mononicotine thiocyanate:					
Cross-striped cabbage worm	8.5	200	3	Moderate	83
	3.4	200	2	Much	0
Hawaiian beet webworm	5.0	200	3	Trace	83
Melon worm	5.0	200	3	Moderate	85
Pickleworm	5.0	190	3	Trace	100
	2.5	205	2	---do---	100
Southern armyworm	3.4	200	2	Much	3

Table 1 -- Continued

Compound and insect	Nicotine content of dust	Deposit of dust per square centimeter	Period of exposure	Extent of feeding	Mortality
	Percent	Micrograms	Days	Percent	Percent
Cupric dinicotinate thiocyanate:					
Hawaiian beet webworm	11.88	200	3	Trace	100
Melon worm	5.0	200	3	Moderate	100
	2.5	210	2	--do--	46
Pickleworm	5.0	195	3	None	100
	2.5	210	2	--do--	100
Southern armyworm	4.75	210	2	Much	0
Southern beet webworm	4.75	200	2	--do--	44
Nicotine paper yellow L (first fraction):					
Blister beetle	27.0	350	3	Moderate	5
Hawaiian beet webworm	13.5	210	3	Trace	93
Melon worm	5.0	200	3	Moderate	60
Pickleworm	5.0	200	3	None	100
	2.5	210	2	Moderate	100
Southern armyworm	6.75	200	2	Much	0
Southern beet webworm	6.75	200	3	Moderate	80
	2.7	195	2	Much	0
Cuprous dinicotinammino thiocyanate:					
Blister beetle	25.0	200	3	None	5
Melon worm	5.0	195	2	Moderate	75
Pickleworm	5.0	190	3	None	100
	2.5	190	2	Trace	100
Southern armyworm	5.0	190	3	Much	21

Table 1-- Continued

Comparative Tests with Commercial Insecticides

Compound and insect	Strength of dust 1/ Percent	Deposit of dust per square centimeter Micrograms	Period of exposure Days	Extent of feeding	Mortality Percent
Barium fluoride:					
Blister beetle	100	230	3	Trace	92
Derris:					
Cross-striped cabbage worm	25	215	3	Trace	98
Hawaiian beet webworm	50	195	3	do	70
Melon worm	25	205	3	do	61
Southern beet webworm	25	200	3	do	87
Lead arsenate:					
Southern armyworm	25	205	3	Trace	94
Pyrethrum:					
Pickleworm	100	190	2	None	100

1/ Figures in second column refer to the strength of the insecticide in the dust. Thus, a dust containing 25 percent of derris has a rotenone content of 1.2 percent.

All the compounds listed in table 1 were at least as good as the recommended insecticides except in tests with the southern armyworm and the blister beetle. None of the nicotine dusts had any control effect on the southern armyworm at the concentrations used.

Cuprous dinicotinamino thiocyanate repelled the blister beetles but did not kill them. Nicotine paper yellow L (first fraction) had no effect on this insect.

Cuprous mononitotine thiocyanate was about as effective as derris against the cross-striped cabbage worm, but the rotenone content of the derris dust was twice that used in the field.

Five of the materials tested on the Hawaiian beet webworm gave higher mortality than the derris dust, which probably was higher in rotenone than the derris used in practical control.

Against the melon worm cupric dinicotine picrate, zinc dinicotine picrate, cuprous mononitotine thiocyanate, and cupric dinicotine thiocyanate, in dusts containing 5 percent of nicotine, were more effective than derris at the strength recommended for field use (1.2 percent rotenone): nicotine paper yellow L (first fraction) and cuprous dinicotinamino thiocyanate were as effective as derris at the same respective strength.

Against the pickleworm all six of the compounds, at both 5 and 2.5 percent nicotine, were 100 percent effective, as was also the pyrethrum. Zinc dinicotine picrate and nicotine paper yellow L, however, allowed more feeding than did the pyrethrum.

Against the southern beet webworm, cupric dinicotine picrate, zinc dinicotine picrate, and nicotine paper yellow L (first fraction), at 6 percent of nicotine, were approximately as good as derris.

Phytotoxicity Tests with Dusts

A glass cylinder 25 inches high and 11 inches in diameter open at both ends was placed over plants in the garden, and a heavy coating of dust particles was allowed to settle on the leaves. Two applications were made 4 days apart, and the results were recorded 8 days after the first application. From 1 to 12 plants of each kind were used in these tests. The plants were covered only at night and during periods of rain. Since in the tests of insecticidal effect, dusts containing 5 percent of nicotine were generally used, it was decided to make the phytotoxicity tests with dusts containing 15 percent of nicotine to provide for a margin of safety. All the compounds at this nicotine concentration were apparently nontoxic to the plants used.

Tests with Spray Residues

Tests were next made to determine the effect of the residues from aqueous sprays prepared from the compounds. Plants were sprayed with suspensions containing from 0.05 to 0.14 percent of nicotine, and excised leaf sections were placed in 9-cm. Petri dishes with insects. Of the insects used in these tests -- the blister beetle, the southern armyworm, the melon worm, and the pickleworm -- only the last two were affected by the compounds. Table 2 gives the data from the results of only those compounds that were effective against either one or both of these two insects. Here again the majority of the most effective compounds contain copper.

Table 2. --- Toxicity of metal-nicotine compounds effective against the pickleworm and the melon worm as compared with derris, when fed foliage containing spray residue in Petri dishes

Compound	Tests against the pickleworm				Tests against the melon worm			
	Nicotine content of spray	Exposure Days	Extent of feeding	Mortality Percent	Nicotine content of spray	Exposure Days	Extent of feeding	Mortality Percent
	Percent	Days		Percent	Percent	Days		Percent
Cupric dinicotinate thiocyanate	0.1	6	Trace	92	---	---	---	---
	.5	6	Much	96	---	---	---	---
Cupric dinicotinate picrate	.12	4	Moderate	100	---	---	---	---
Zinc dinicotinate picrate	.12	6	---do---	88	---	---	---	---
Nicotine chrome orange R	.1	6	Trace	100	0.1	6	Trace	100
Nicotine paper yellow L	.1	6	---do---	79	.09	6	Moderate	92
Nicotine paper yellow L (second fraction)	.05	6	Moderate	92	---	4	---do---	100
Cupric nicotine paper yellow L	---	---	---	---	.14	---	---	---
Cupric nicotine stilbene yellow GA	.08	6	---do---	100	---	4	---	100
Cuprous mononicotinate thiocyanate	.1	6	Trace	88	.085	---	---	---
	.05	6	Moderate	88	---	---	---	---
Cobaltous dinicotinate thiocyanate	.05	4	---do---	83	---	---	---	---
Nickelous dinicotinate thiocyanate	.05	6	---do---	96	---	---	---	---
Cuprous nicotine cyanide	0.1	6	Trace	100	0.1	4	Moderate	100
	.05	6	Moderate	88	---	4	---do---	96
Cuprous dinicotinamino thiocyanate	.1	4	Trace	100	.12	---	---	---
	.05	4	Moderate	100	---	4	Trace	94
Derris	0.026/1	4	Trace	69	---	6	---do---	100
	---	6	---do---	84	---	---	---	---

Rotenone content.

At the concentrations used all the compounds listed in table 2 that were tested against the pickleworm were equal to derris, but in several instances moderate feeding was evident. The six compounds found to be effective against the melon worm were also superior to derris, with the possible exception of nicotine paper yellow L (second fraction).

Phytotoxicity Tests with Spray Residues

Phytotoxicity tests were made on 5 kinds of plants in the garden plot by thoroughly spraying them with suspensions of the various nicotine compounds containing approximately 0.3 percent of nicotine, 3 times the strength used in the insect tests. At this concentration only 2 of the 12 compounds that showed insecticidal properties when applied as sprays had no effect on foliage. They were cuprous nicotine cyanide and cuprous dinicotinammino thiocyanate. When the amount of nicotine was reduced to twice that used in the insect tests, only two other compounds, cupric nicotine paper yellow L and cuprous mononitotine thiocyanate, were added to the list of materials safe to use. All the others caused from moderate to severe injury to 2 or more kinds of plants.

Contact Tests with Sprays and Dusts

Tests were made on red spiders found on celery in the field by thoroughly dusting them with a small hand duster and setting the stems of the foliage in flasks of water in battery jars. By the method used it was impossible to determine the amount of dust deposited. Of 24 materials tested, only 2 -- cupric dinicotine benzoate (39 percent mortality) and cuprous nicotine cyanide (52 percent mortality) -- showed any toxicity in 3 days. In the same period dusting sulfur effected 100 percent mortality.

Spray tests against the aphid Macrosiphum ambrosiae were made with 19 of the compounds diluted to 4 pounds per 100 gallons of water, which resulted in nicotine concentrations from 0.08 to 0.25 percent, with 1 percent of saponin added as a wetting agent. The following compounds caused at least 89 percent mortality (percentages of nicotine in parentheses): Cupric dinicotine benzoate (0.185), cupric dinicotine salicylate (0.173), zinc dinicotine salicylate (0.173), zinc dinicotine benzoate (0.185), cupric nicotine chrome orange R (0.088), cupric dinicotinammine benzoate dihydrate (0.243), cupric dinicotinammino o-benzoylbenzoate dihydrate (0.186), and cuprous dinicotinammino thiocyanate (0.250). Nicotine sulfate (40 percent nicotine) diluted to 0.04 percent in water caused 100 percent mortality. Saponin alone caused 4 percent mortality. The other compounds gave mortalities ranging from 0 to 69 percent. The following chemicals were not included in this test because they were too toxic to the plants at the concentration used: Cupric dinicotine thiocyanate, zinc mononitotine thiocyanate, cobaltous dinicotine thiocyanate, nickelous dinicotine thiocyanate, manganese mononitotine salicylate, and cadmium dinicotine salicylate.

Another test gave some indication of the contact action against lepidopterous larvae. Insects in Petri dishes were dusted at the rate of approximately 200 micrograms per square centimeter and fed untreated foliage. By this method it was possible for the insects to pick up and eat particles of insecticide from the bottom of the dish. At the end of 2 and 3 days mortality counts and estimates of the amount of feeding were made. Against the melon worm, with the use of 10 percent dusts (nicotine 1.6 to 5.25 percent) in not one case was the material so efficient as in the dusted-foliage test with the same amount of nicotine. Against the southern army-worm, however, when the undiluted material was used the following compounds were at least as effective as in the dusted-foliage tests: Cupric dinicotine thiocyanate, cupric dinicotine benzoate, zinc mononitotine thiocyanate, cuprous mononitotine thiocyanate, cuprous dinicotine thiocyanate, cobaltous dinicotine thiocyanate, cuprous nicotine cyanide, and cadmium dinicotine salicylate.

Miscellaneous Tests Against Roaches, Weevils, and Termites

All the materials were tested against the American cockroach by sprinkling 0.25 gm. over the bottom of a 6-inch battery jar and then introducing 10 nearly full grown nymphs. A thin film of petrolatum was put on the top inner surface of the jar to prevent the escape of the insects. Cuprous nicotine cyanide was the only effective material, causing 100 percent mortality in 3 days. An equal weight of sodium fluoride, however, killed all the roaches in 1 day.

In tests against the cowpea weevil each compound was thoroughly mixed with Brown Crowder cowpeas, 1 part to 1,000 parts by weight, in a Petri dish. Thirty adult weevils were then introduced, and each dish was placed on its edge. After 4 days mortality counts were made. None of the materials caused a mortality greater than 50 percent. The five that killed from 40 to 50 percent were cupric dinicotine benzoate (49 percent mortality), cuprous nicotine cyanide (48 percent), cupric dinicotinammino benzoate dihydrate (44 percent), zinc mononitotine thiocyanate (43 percent), and nicotine chrome orange R (42 percent).

The effectiveness of the materials against termites was determined by the method described by Hockenyos. ^{5/} A small piece of crumpled tissue paper and approximately 15 ml. of water were placed in the bottom of a 100-ml. beaker. On top of this a 40-gm. mixture of the compound and sand was introduced. After all the sand had become damp, approximately 30 adults or large nymphs were placed in the beaker. Mortality counts were made at the end of 4 days. Cupric dinicotine benzoate killed 56 percent of the insects at a dilution of 1 to 1,000 and 22 percent at 1 to 5,000, cuprous nicotine cyanide at 1 to 1,000 repelled the insects and gave a mortality of 15 percent but was ineffective at 1 to 5,000, and cupric dinicotinammino o-benzoylbenzoate dihydrate repelled the insects and killed 54 percent of them at 1 to 1,000, but was ineffective at 1 to 5,000. None of the other compounds caused any mortality.

^{5/} Hockenyos, G. L. Laboratory evaluation of soil poisons used in termite control. Jour. Econ. Ent. 32:147-149. 1939.

Summary

Several types of tests were made to determine the insecticidal efficiency of 25 complex salts containing nicotine, usually combined with a metal.

In feeding tests with dusts diluted to approximately 5 percent nicotine content, at least six of the compounds were as effective against two to four species as was the recommended insecticide. These compounds, in descending order of effectiveness against from four to six species, were cupric dinicotine picrate, zinc dinicotine picrate, cuprous mononitotine thiocyanate, cupric dinicotine thiocyanate, nicotine paper yellow I (first fraction), and cuprous dinicotinamine thiocyanate. When these materials were applied to plants in dusts containing 15 percent of nicotine, little or no injury to the foliage was apparent.

When tested as sprays, 12 of the compounds were as effective as the recommended insecticides against at least 1 of 4 species of insects on foliage sprayed with suspensions containing approximately 0.1 percent of nicotine. Of these 12 only 2, cuprous nicotine cyanide and cuprous dinicotinamine thiocyanate, caused no injury to foliage in spray suspensions containing 0.3 percent of nicotine. These are, therefore, the only compounds that can be recommended as safe to use in spray form.

Most of the compounds that showed appreciable toxicity against lepidopterous larvae contained copper. Only two materials, cuprous mononitotine thiocyanate and cuprous dinicotinamine thiocyanate, were 100 percent effective in both dust and spray form.

When dusted directly in Petri dishes and fed untreated foliage, melon worm larvae were only slightly affected by any of the materials, but the southern armyworm was affected by several where it was possible in each test for the larvae to eat some of the particles.

Cuprous nicotine cyanide killed all the large nymphs of the American cockroach in 3 days. None of the materials were effective against the cowpea weevil or against termites.